



## **Revised Intumescent Coatings for the 60-mm PA124 Military Container**

**by Pauline Smith and Kestutis Chesonis**

**ARL-TR-3679**

**November 2005**

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# **Army Research Laboratory**

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## 1. Introduction

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An intumescent coating system for fire and heat protection is specified for 60-mm mortar containers. The projected intumescent coating system for ammunition containers performed unsuccessfully during testing at the Aberdeen Test Center, Aberdeen Proving Ground, MD. This project revealed a number of concerns including paint delamination, cracking, and chipping after the drop and impact-resistant test. The specified intumescent system (intumescent and topcoat) was supplied by NoFire Technologies Inc. and consists of a water-based A-18NV, MIL-PRF-24596 (1999), type II, intumescent paint and a solvent-based topcoat (Uracoat<sup>\*</sup>). The intumescent coating system is applied over the forest-green coating (basecoat) used on the containers.

Testing performed on the NoFire intumescent coating system has shown it has poor flexibility, poor impact characteristics, and marginal moisture resistance. In order to improve the intumescent coating system flexibility, a water-based emulsion topcoat was formulated by the U.S. Army Research Laboratory (ARL) to replace the Uracoat. The water-based emulsion topcoat, when applied over the NoFire intumescent coating, showed improved flexibility and impact resistance, but performed poorly in moisture resistance tests. Also, the same tests were performed on the NoFire intumescent with various Army coatings (i.e., MIL-DTL-64159 [2002] chemical agent resistant coating [CARC] topcoat, MIL-DTL-11195E [2003], type II, and ARL water-based emulsion topcoat) and showed similarly poor results as the Uracoat. As a result, it was concluded that the NoFire intumescent coating with any topcoat was designed for interior use, and an alternative intumescent coating system was pursued.

PPG Industries Inc. supplied epoxy-based Firetex<sup>†</sup> M78 intumescent coating samples for comparison to the current intumescent latex-based coating system. PPG Industries Inc. also supplied separate sets of panels with two other intumescent coating samples: Pitt-Char LV<sup>†</sup> (epoxy based) and M78 Firetex (alkyd based). Both sets of PPG supplied panels were coated with the Pitt-Tech topcoat.<sup>†</sup>

To test the coating for its heat protection properties, burnt tests were performed using a propane torch on the coating systems. Initial results show the Pitt-Char LV system performed poorly, but the M78 Firetex system performed as well or better than the NoFire control system. The preliminary moisture resistance tests show the Pitt-Char LV has no significant improvement over the NoFire, but the M78 Firetex has much better moisture resistance than the NoFire. As a result, additional testing of the M78 Firetex intumescent coating system will be pursued, using current Army topcoats, MIL-E-11195E, type II, and MIL-C-53039B (2005) as replacement topcoats for the intumescent coating system.

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<sup>\*</sup>Uracoat is a registered trademark of NoFire Technologies Inc., Upper Saddle River, NJ.

<sup>†</sup>Firetex, Pitt-Char LV, and Pitt-Tech are registered trademarks of PPG Industries Inc., Pittsburgh, PA.

The coating currently used as a control sample by the U.S. Army Armament Research, Development, and Engineering Center (ARDEC) on ammunitions containers was supplied by NoFire. The NoFire A-18NV, MIL-PRF-24596, type II, intumescent coating is a one-part water-based coating that requires a topcoat for weather-resistant finish, usually applied in two stages to meet the substantial dry film thickness (DFT) requirement. Comparatively, the epoxy-based Intumescent coating was supplied by Leigh's Paint Co., for which PPG is the manufacturer's U.S.A. representative; the coating was applied by brushing onto the cured basecoat.

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## **2. Objectives**

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It is proposed to use a three-part coating system in order to comply with the protection requirements for the 60-mm mortar round containers. This system will consist of a primer, an intumescent coating, and a topcoat. The ultimate objective of this project is to address the difficulties identified in earlier testing, which are mainly problems of flexibility, impact resistance, and moisture resistance. One of these products to be evaluated and qualified is the PPG intumescent coating. Additionally, it will also be necessary to evaluate and qualify a topcoat for the intumescent coating. The process of applying these three coatings will also be evaluated for improvement at a nominal cost and minimal impact on application.

Three approaches will be considered to address these issues. The first approach will be to evaluate PPG's Firetex M78 intumescent coating by comparing it to the current intumescent coating system while using existing military topcoats such as MIL-DTL-11195E (proposed type II) or MIL-DTL-64159 as replacements for current topcoat. A second approach will be to implement a "tie" coat for system modification, while the third approach would be to add modifiers to increase the surface profile of the system.

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## **3. Experimental**

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### **3.1 Qualification Test**

ARL and ARDEC will evaluate various topcoats with M78 Firetex intumescent coating. Evaluation of the topcoats will include the following tests: flexibility, adhesion, impact, humidity, and water immersion. In addition to the tests performed on just the topcoat, burn tests will be performed on the intumescent/topcoat system.



### 3.2 Coating Description

The system consists of three unique coatings:

1. Basecoat – Brockway’s alkyd basecoat (paint for ammunition containers), applied by dip-coat directly to the substrate, then baked at 350 °F for 10 min with a DFT of 1–2 mil.
  2. Subsequently, an intumescent coating is applied by airless spray or by brushing onto the cured basecoat.
  3. Finally, the intumescent coating is top-coated primarily to provide a barrier for high resistance to severe weather, abrasion, and corrosive chemicals or fumes.
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## 4. Coating Materials to Be Evaluated

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- MIL-DTL-11195E, type II, is fast curing, lusterless, corrosion resistant, alkyd enamel for use as a single coat for the medium and large caliber ammunition system; it is nonreactive and compatible with the explosive in the munitions.
- MIL-C-53039B is a one component aliphatic polyurethane CARC for use as a finish coat on military equipment.
- MIL-DTL-64159 – water-dispersible aliphatic polyurethane CARC.
- Uracoat – two component aliphatic urethane enamel supplied by NoFire.
- Pitt-Tech, supplied by PPG High Performance Coatings, is an acrylic waterborne, high-gloss enamel that provides corrosion protection, chemical and solvent resistant, satin fast-drying finish.
- No-Fire Technologies Inc. A-18NV, MIL-PRF-24596, type II, is a water-based intumescent paint.
- Leigh’s M78 Firetex epoxy – solvent-based intumescent coating, is a thin film, single component material that will provide up to 2-hr fire protection of structural steel when used as the intumescent coating replacement.
- Pitt-Char LV, supplied by PPG Architectural Finishes, is a two component, low viscosity epoxy-based intumescent coating. This durable epoxy coating will transform into a charred ceramic-like insulator material providing thermal protection.
- Speedhide\* – low gloss, water-based acrylic latex.

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\*Speedhide is a registered trademark of PPG Industries Inc., Pittsburgh, PA.

- ARL water-based emulsion – ARL formulated water-based emulsion topcoat.
  - Sherwin-Williams Company, Kem Aqua<sup>\*</sup> 70P, a waterborne, fast-drying, moisture resistant, corrosion resistance metal primer which is used as the “tie coat” material. It was applied at 0.3–0.6 mil dry film and allowed to air dry for 45–60 min prior to applying M78 Firetex.
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## **5. Technical Approach**

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This effort consists of evaluating various topcoats with the M78 Firetex intumescent coating in a laboratory environment. The laboratory testing will be conducted at the ARL and ARDEC facilities using standard cold-rolled steel panels coated with the container basecoat. Concurrently, DZI/PC Boards will conduct application field-testing of the M78 Firetex intumescent with various topcoats on 60-mm containers to ensure technical viability. The coated containers will be sent to ARL for testing and evaluation. Also, an investigation will be conducted on changing the pigmentation on the M78 Firetex from white to the forest green specified by the container drawings.

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## **6. Experimental**

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For this project, impact, flexibility, and cross-hatch adhesion test were used to characterize and identify the best performers. To determine performance improvement, the new data will be compared to baseline data from earlier testing. The coating application process was evaluated to develop feasible production process parameter guidelines while optimizing curing time and temperature.

### **6.1 Mandrel Bend Test (Flexibility)**

The cylindrical mandrel bend test was performed on all coatings in accordance with ASTM D 522–93 (1993) to determine resistance to cracking.

### **6.2 Impact Resistance**

The impact resistance test was performed on the candidate coatings based on ASTM D 2794 (2004). To determine impact or rapid deformation of a coated film, a standard weight is dropped a specified distance so as to strike an indenter that deforms the coating and possibly the substrate. The indentation and visible cracks determine whether the sample is acceptable or is a failure.

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<sup>\*</sup> Kem Aqua is a registered trademark of the Sherman-Williams Company.

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## 7. Sample Preparation

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The systems were prepared as specified in this section.

### 7.1 Control System

- Brockway's alkyd basecoat was applied by the dip method and baked at 350 °F to achieve a DFT of 1.0–1.5 mil.
- Leigh's epoxy-based intumescent coating, M78 Firetex was applied by brush and air dried at room temperature to achieve a DFT of 25–35 mil.
- Topcoated with MIL-DTL-11195E, type II, or MIL-C-53039B, applied using High Volume Low Pressure (HVLP) spray method and ambient cured to achieve a DFT of 1.0–1.5 mil.

### 7.2 System With a Tie Coat

- Brockway's alkyd basecoat was applied by the dip method and baked at 350 °F to achieve a DFT of 1.0–1.5 mil.
- Sherwin-Williams Company, Kem Aqua 70P was applied by brush and air dried at room temperature to achieve a DFT of 0.5–0.7 mil.
- Leigh's epoxy-based intumescent coating, M78 Firetex was applied by brush and air dried at room temperature to achieve a DFT of 25–35 mil.
- Topcoated with MIL-E-11195E, type II, or MIL-C-53039B, applied using HVLP spray method and ambient cured to achieve a DFT of 1.0–1.5 mil.

### 7.3 Scuff Basecoat

- Brockway's alkyd basecoat was applied by the dip method and baked at 350 °F to achieve a DFT of 1.0–1.5 mil.
- Brockway's alkyd basecoat was applied by the dip method and baked at 350 °F to achieve a DFT of 1.0–1.5 mil. The basecoat was then scuffed with Scotch-Brite.\*
- Intumescent coating M78 Firetex was applied by brush and air dried at room temperature to achieve a DFT of 25–35 mil.
- Topcoated with MIL-E-11195E, type II, or MIL-C-53039B, applied using HVLP spray method and ambient cured to achieve a DFT of 1.0–1.5 mil.

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\* Scotch-Brite is a registered trademark of 3M, St. Paul, MN.

#### **7.4 Sanded Basecoat**

- Brockway's alkyd basecoat was applied by the dip method and baked at 350 °F to achieve a DFT of 1.0–1.5 mil.
  - Brockway's alkyd basecoat was applied by the dip method and baked at 350 °F to achieve a DFT of 1.0–1.5 mil. The basecoat was sanded with 325-grade paper.
  - Intumescent coating M78 Firetex was applied by brush and air dried at room temperature to achieve a DFT of 25–35 mil.
  - Topcoated with MIL-E-11195E, type II, or MIL-C-53039B, applied using HVLP spray method and ambient cured to achieve a DFT of 1.0–1.5 mil.
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### **8. Observations**

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The panels prepared as previously specified were subjected to a controlled environment of 100% humidity at 100 °F for 48 hr. An additional set of panels were immersed in water for 48 hr at room temperature. After exposure the panels were bent using a 1-in mandrel bend and tested for flexibility.

After flexibility testing, MIL-DTL-11195E (no. 1) demonstrated film popping and delaminating from the basecoat, while the MIL-C-53039B (no. 1) developed minor cracks.

The same systems were later tested for integrity. Firmly cutting with pressure using a sharp knife edge on the stressed area, the MIL-DTL-11195E resisted any lateral displacement by the knife edge; however, the coating could be easily displaced. The MIL-C-53039B coatings demonstrated better resistance to the knife edge but had the tendency to pop off. The systems were further examined for adhesion between the basecoat and Firetex by incorporating a tie coat and by means of scuffing or sanding the basecoat using Scotch-Brite and 325-grade sand paper.

The systems that involved the tie coat (no. 2) or scuff (no. 3) and sanding (no. 4) immeasurably improved the flexibility and adhesion of the overall system. MIL-DTL-11195E passed the full flexibility bend, had moderate cracks, while the MIL-C-53039B passed with finer cracks; however, both showed improved adhesion to the basecoat by showing no sign of delamination and no visible cracks to the substrate or basecoat.

Based on these results, scuff and/or sanding the basecoat to reduce the gloss or using a tie coat will definitely provide an overall improved system. No improvement was observed in the results when using sand paper or Scotch-Brite.

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## 9. Humidity and Water Immersion

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After subsequent exposure to water immersion and humidity testing, all systems showed some loss of adhesion to the basecoat. Overall, the MIL-C-53039B coatings showed moderately better adhesion than the MIL-DTL-11195E. The basecoat by itself was a good, tough coating, but as a combined system it was too glossy and smooth to hold the thick adhering coatings.

For comparison, four samples each of the Pitt-Char LV and Leigh's M78 Firetex were tested, with and without the PPG Pitt-Tech topcoat. Two of each sample went into the cabinet at 100 °F and at 100% humidity. They were checked every 24 hr for 3 days, noting any evidence of softening or blistering.

The Pitt-Char LV two-component epoxy samples had an average DFT of 27–34 mil applied directly to the bare metal. After 24 hr of exposure, the coating showed softening of the film. After 48 hr, the coating began to pop and could be easily and severely damaged by a blunt spatula with moderate pressure applied. After 3 days, the coating was easily removed from the substrate.

The Pitt-Char LV, in combination with PPG Pitt-Tech topcoat, was applied at 2 mil DFT and was noticeably tacky to touch prior to exposure in the chamber. The coatings were malleable after 24 hr and the spatula could easily touch down to the metal; it was even softer after 48 hr. After 3 days, small portions of the coating could be removed easily from the metal. After allowing a 4-hr recovery period, at room temperature and humidity, the system was reevaluated and noted that the coating did not show any signs of delamination of the topcoat as a unit when bent to test for flexibility.

The Leigh's M78 Firetex sample had an average DFT of 26–34 mil applied directly to the bare metal. After 24 and 48 hr of exposure, these coating were fairly tough, showing no softening of the film. After 3 days of exposure, the coating was tough and intact; however, using the spatula with moderate pressure penetrated to the metal substrate. The Leigh's M78 Firetex and PPG Pitt-Tech topcoat samples were also intact after 48 hr; after an additional 24 hr, using the spatula with moderate pressure penetrated to the intumescent layer. Further evaluation showed that the M78 Firetex intumescent and Pitt-Tech topcoat did not delaminate after bending but did show short cracks. This indicates that the topcoat has good adhesion to the intumescent paint but tended to delaminate when stressed.

The Pitt-Char LV coated samples were subjected to water immersion and after a mere 4 hr showed signs of softening but no signs of blisters. Following subjection to water immersion for 8 hr, noticeably large blisters were on the total immersed areas.

The M78 Firetex, after subjected to water immersion for 4 hr, showed slight loss of adhesion, and after 8 hr, the film had softened but remained intact.

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## **10. Weight Loss**

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The intumescent coating application, if inadequately cured, will maintain residual water, causing poor adhesion; likewise, humidity and heat have a tendency to eliminate most of the water residuals in the intumescent coating. It is not possible to speed up rate of dry or cure of M78 Firetex. It basically hardens merely by losing solvent and unfortunately some of the resin tends to hang onto the solvent; the current solvent blend gave the fastest drying time. Since the intumescent coat was applied densely at 9–15 mil DFT, this thick film will allow entrapments of water even after application of the topcoat. The intumescent coating was applied onto bare metal and also to basecoat and allowed to air dry to see if and how the solvents evaporate as a function of time and curing temperature. Three panels were coated with only the M78 Firetex intumescent coating (directly to metal) and subjected to 170 °F. The weight was monitored at regular time intervals (5, 10, and 20 min); the test was terminated either when the coating was no longer soft to the touch or when the weight loss was marginal.

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## **11. Application Procedure**

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- Sample A: applied intumescent coating and force dried at 140 °F for 30 min, followed by ambient air fan cooling for 3 min.
- Sample B: applied intumescent coating and force dried at 170 °F for 30 min, followed by ambient air fan cooling for 3 min.
- Sample C: applied intumescent coating and force dried at 190 °F for 30 min, followed by ambient air fan cooling for 3 min.
- Sample D: applied intumescent coating and air dried for 24 hr.
- Samples A, B, C, and D were allowed an additional 30 min at 140 °F to ensure full curing. The data showed no additional change in weight loss. It is interesting to note that 30–35 mil DFT was obtained in one application.

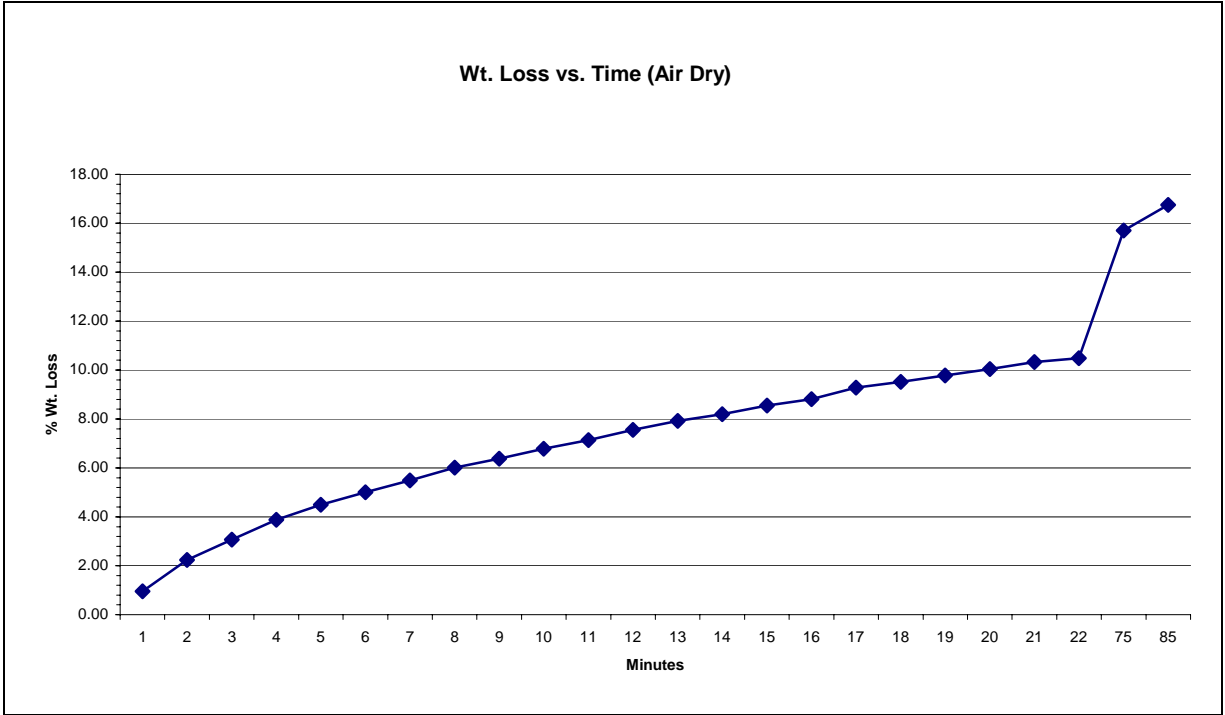
After reviewing the data and based on these results, air drying for a minimum of 16 hr or force drying at 140–170 °F for a minimum of 45 min is recommended.

Figures 1 and 2 show the weight loss of a cured coating over time.



Note: 0.043 in WFT @ 140.

Figure 1. Weight loss vs. time at 140 °F.



Note: 0.043 in WFT.

Figure 2. Weight loss vs. time at ambient temperature.

While there is no hard data to reflect that the product was fully cured, it was observed with a “fingernail test” that the 0.043-in coating was not fully cured until 72 hr after removal from the oven. The 0.022-in coating was not fully cured until 8 hr after removal. The 0.015-in coating was fully cured after ~30 min. The coating needs to be fully cured in order to provide accurate thickness measurements; therefore, a multiple coat application with force drying between coats is recommended.

Figure 3 shows samples 1, 2, and 3 were baked for 30 min at 140 °F, air dried for 16 days, and thereafter forced dried for 30 min at 140 °F. After 5 additional days, the samples showed constant weight and were unaffected by heating.

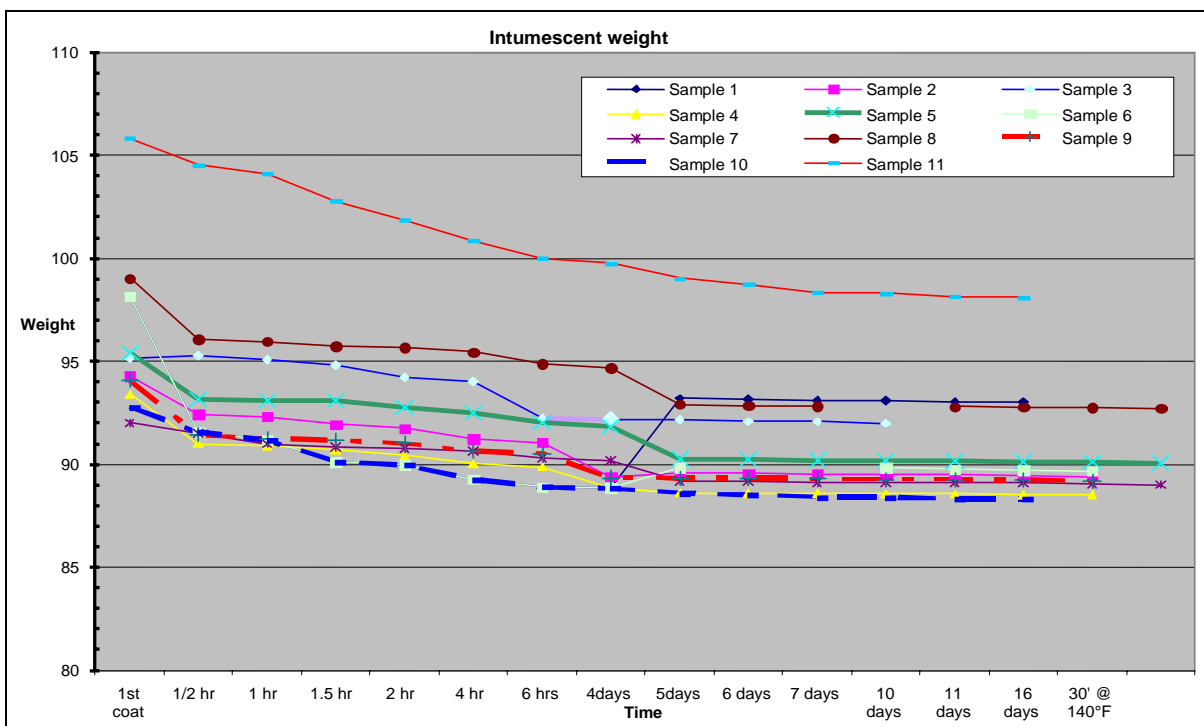


Figure 3. Weight loss vs. time at ambient temperature, in addition to 30 ft at 140 °F.

Samples 4, 5, and 6 were baked for 30 min at 170 °F, air dried for 16 days, and thereafter forced dried for 30 min at 140 °F. After 5 additional days, the samples showed constant weight and were unaffected by heating.

Samples 7, 8, and 9 were baked for 30 min at 190 °F, air dried for 16 days, and thereafter forced dried for 30 min at 140 °F. After 5 additional days, the samples showed constant weight and were unaffected by heating.

Samples 10 and 11 were air dried for 13 days and thereafter forced dried for 30 min at 140 °F. After 7 additional days, the samples showed constant weight and were unaffected by heating.



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## 12. Material Testing

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PC-Boards in Kansas coated PA124 containers with Firetex, M78 intumescent coating. DFT was the acquired thickness in one single application; however, the drying time was quite lengthy due to the high solvent content and rapid “skinning over” which impedes the solvents from outgassing. The containers were preprimed with Kem Aqua bonding primer, then topcoated with the aliphatic polyurethane CARC, MIL-C-53039B. The intumescent coating was applied between the primer and the topcoat.

One of the containers had the original basecoat scuffed with a 3M Scotch-Brite pad prior to the intumescent paint application.

All painted containers were dried overnight in a paint booth with the vent activated, and all seemed dry to the touch the next morning. However, subsequent checks revealed that the intumescent coating had not completely cured. These checks also revealed that the proper thickness had not been obtained for some areas on each container.

For material testing we received three containers prepared by PC-Boards in Kansas. The containers were the standard 60-mm mortar containers, prepared and finished as specified with the following combinations:

- Sample 1: NoFire, control system  
TDP basecoat (Brockway alkyd), intumescent (MIL-PRF-24596, type II, NoFire A-18NV), Uracoat, a 2K urethane topcoat.
- Sample 1P: M78, system with a tie coat  
TDP basecoat (Brockway waterborne alkyd), Kem Aqua WR bonding primer, Leigh’s M78 Firetex II, and MIL-C-53039B.
- Sample 1B: M78, system with scuffed basecoat (Scotch-Brite)  
TDP basecoat (Brockway waterborne alkyd), basecoat scuffed with Scotch-Brite, Leigh’s M78 Firetex, and MIL-C-53039B.

Material testing is accomplished by placing the materials through arduous tests to see how long the system endures before breaking down. In essence any coating is reliably expected to stand up to the wear and tear of its target application. For this project, the containers were initially subjected to 100% relative humidity. The complexity of this project includes evaluating paint delamination, cracking, and chipping after the drop and impact-resistant test.

## **12.1 Humidity**

The coated containers were placed in an enclosed chamber surrounded by a heated, saturated mixture of air and water vapor. The temperature of the chamber was maintained at 100 °F and 100% relative humidity. The concept process is that the water will permeate the coating at rates that are dependent on the characteristics of the coating.

## **12.2 Impact Resistance**

The standard impact resistance test was performed on the candidate coatings based on ASTM D 2794.

## **12.3 Observations**

To evaluate system failure analysis/performance improvement, stress tests were employed to quantify adhesion and cracking problems. These tests included impact, flexibility, and dropping the container with 25 lb of weights. To evaluate impact or rapid deformation of a coated film, the weight was dropped a distance so as to strike an indenter that deforms the coating and possibly the substrate. This indentation and visible cracks are then evaluated to determine the failure.

After being exposed to 100 °F and 100% relative humidity for 24 hr, and immediately after removal from the humidity cabinet, the containers were tested for system integrity. Firm cutting pressure by a round-edge spatula showed that both samples coated with the M78 system were fully protected. They were slightly softened and resisted any lateral displacement by the flat side of the spatula. The system with the NoFire A-18NV, MIL-PRF-24596, type II, coating was highly softened and could be displaced by fingernail and easily displaced with the spatula down to the basecoat.

After allowing 4 hr of recovery, the results were similar for all three containers. Following 168 hr at 100 °F and 100% relative humidity, the results were comparable to the previous exposure. Consequent to exposure to humidity, water had permeated and softened the container painted with the NoFire A-18NV, MIL-PRF-24596, type II, coating.

Prior to conducting further testing, the containers were baked in a convection oven for 4 hr at 100 °F and then allowed to cure in ambient air.

## **12.4 Resistance Test**

To improvise an impact test on the containers, we dropped a dummy 120-mm mortar from a table top (~3 ft) onto the containers at ground level. The NoFire A-18NV, MIL-PRF-24596 sample chipped and delaminated to the bare metal after the drop. The M78 sample showed chips and cracks to the basecoat and significant improvements with the use of the “tie” coat. Essentially, after dropping the dummy 120-mm mortar onto the container, the indentation on the containers was very inconsistent and indistinct.

Therefore, to expand on the drop test, a 25-lb weight was added to the inside the container and dropped from the table top. To evaluate impact or rapid deformation of the coated film, the weighted container dropped a distance, so as to strike an indenter that deforms the coating and possibly the substrate. The indentation and visible cracks determined the failure was similar to the previous test. Figure 4 shows the results of paint chipping and cracking.

Parts of the flexibility and adhesion were evaluated utilizing a procedure in which the containers were subjected to moderate pressure and pounding with a hammer. The results were similar to previous data on the laboratory-prepared samples and complemented the test data.

The containers were later sectioned into parts that enabled easier handling and testing for flexibility and for impact testing. After bending at  $\sim 1/2$  in, sample 1P (M78 with tie coat) showed major improvements over all samples with very small cracks. Sample 1B (M78, Scotch-Brite) and sample 1 (control, NoFire) were equal in the extent of fracture with delamination and cracking to the basecoat.

Sample 1P and sample 1 had relatively high impact resistance, after 40 in-lb impact, the results showing only visible cracks, while sample 1B had severe cracking and paint damage.



Figure 4. The three containers after evaluations.

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## 13. Results

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The impact-resistant test, performed on all the coatings, demonstrated that the M78-coated containers showed signs of cracking to basecoat, whereas cracking on the NoFire was evident quite easily through the entire thickness of coating, revealing bare metal on the NoFire container. The optimal coating was the M78 with the tie coat, as in sample 1P. This coating was not penetrated to a great extent as the others and therefore sealed the cracks and moisture for the very thick intumescent coating. This sealing property provides good adherence to the intumescent coat. To achieve the 30–40 mil DFT, and given that cure time is a function of thickness, the intumescent coating should be applied in at least two coats. This paint appears to cure from the outer coats towards the inner coats, primarily due to the resins releasing the solvents. Therefore, the thicker the coat, the longer it takes the solvents ( $30\% \pm 4\%$  by weight) to outgas.

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## 14. Development Work

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- Evaluate an epoxy intumescent coating in conjunction with an optimum top-coat.
- Optimize application time, temperature, and effects of film thickness variations.
- Develop and incorporate production formulation.
- Manufacture paint sample for demonstration.
- Monitor production trials.

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## 15. References

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